DESIGN AND SCALE-UP OF VACUUM DISTILLATION PROCESS FOR THE DE-ACIDIFICATION OF CRUDE PALM OIL.

1. SYNOPSIS.

The current predicament faced by Malaysian palm oil refiners is the unavailability of the basic performance parameters of structured packings e.g. H.E.T.P., in the physical refining of crude palm oil. Existing production columns cannot measure performance parameters due to its inherent design and lack of instrumentation. Hence, there is a need to develop an indigenous capability to pursue and develop solutions of this problem.

This paper relate the rationale, principles and on -- going development of such a project. The joint research project involve UNIVERSITI TEKNOLOGI MALAYSIA, FELDA REFINING MALAYSIA and SULZER CHEMTECH SINGAPORE.

The aim of this project is to investigate the performance of the structured packings manufactured by SULZER CHEMTECH for the refining of Malaysian crude palm oil by FELDA REFINING MALAYSIA to improve the process efficiency, palm oil quality and product flexibility of the existing plant in PANDAMARAN, Port Klang.

Existing structured packing correlation have being tested for operating conditions of the minimum of 25 torr pressure and below 200 °C temperature. These comparative experiments were also limited to standard distillation mixtures. These tests have revealed that these correlations do not predict well for very low pressure conditions. Furthermore, it has been not being tested for materials with low surface tension i.e. below 40 mN/m2.

To predict structured packing performance for edible oils refining, tests must be conducted on high molecular weight, temperature sensitive material which must be distilled at pressure at low vacuum and high temperature. Typical refining columns operate at the temperature range between 150 °C to 250 °C and pressure below 2 torr.These conditions are beyond the existing experimental operating conditions. In addition, this type of material also possess very low surface tension i.e. below 40 mN/m2 since surface tension lowers as molecular weight increases. Alternative methods such as the extrapolation of existing laboratory and pilot plant results are not reliable since properties changes non-linearly at these extreme conditions.

The scope of work include the initial development of a structured packings laboratory rig of diameter 25 mm and the process software to model and predict the basic H.E.T.P. of the process at conditions similar to that encounter in the physical refining.

Consequently, a pilot plant of diameters of 80mm, 160mm and 240 mm column will be used to investigate the changes of H.E.T.P. due to scale-up factors.

This comparison between the laboratory based simulation of the H.E.T.P. and the experimental pilot plant H.E.T.P. data will determine whether current structured packing theories and correlations can be apply for scale-up and design of production scale refineries.

If not so, modifications will be propose to the existing techniques to improve their accuracy and applications in palm oil processing.

The project has yielded several new areas of study on the palm oil. The physio-chemical properties required for the prediction model is being determined from experiments on surface tension, diffusivity and vapour liquid equilibria. These exceptional equipment operate at very high temperature, up to 300 °C, and very low vacuum pressure, 0.0001 torr.

The research project has successfully obtained softwares to assist in the process design, modelling and simulation of structured packing vacuum distillation pilot plant operations. Nevertheless, H.E.T.P. prediction programs using various correlations under test, Bravo (1990), Messner & Cheng (1985), Spiegel & Meir (1987) and Billet (1992), are being written.

Next, a laboratory scale experiment rig has been built to measure the basic H.E.T.P. of the vacuum distillation of free fatty acids from crude palm oil. Basic H.E.T.P. is defined as the performance not influenced by scale-up factors, Hufton & Bravo (1985).

In conclusion, the project has demonstrated the feasibility of a collaborative research between the private sector and academia once mutual ideals, goals, means and limitations are understood and forged. This is vital for Malaysian refiners to make the best decisions on palm oil processing technology. The best technology will ensure that Malaysian palm oils will be competitive in the global edible oils market of the future.

2. AIM.

The aim is to investigate the performance of the structured packings manufactured by SULZER CHEMTECH in the refining of Malaysian crude palm by FELDA REFINING MALAYSIA to improve the process efficiency, palm oil quality and product flexibility of the existing plant in PANDAMARAN, Port Klang.
(refer figure 1).

3. INTRODUCTION.

1 Process Design Constraints.

The existing constraints in Malaysia of making process design improvements to increase the efficiency, quality and flexibility of the vacuum distillation of crude palm oil de-acidification process are:-

- The lack of the basic physio-chemical properties for the calculation of the structured packings performance parameter, H.E.T.P, in the vacuum steam distillation of crude palm oil i.e. physical refining of crude palm oil. This is defined as infra-structure development.

- The lack of indigenous capability to computer design, model and simulate of new process changes in order to evaluate its operational and economical impact. This is defined as process development.

- The lack of indigenous capability to conduct scale - up study of laboratory results up to pilot plant scale operations to yield commercial value results.This is defined as product development.

2 Areas of Research.

1. Physio-chemical Properties.

The physio-chemical properties shortcoming is being overcome with the fabrication of critical properties measurement rigs which are used for structured packings design calculations. These are for measuring surface tension, diffusitivity and vapour liquid equilibria of palm oil mixtures. These exceptional equipment operate at very high temperature, up to

300 °C, and very low vacuum pressure, 0.0001 torr. These are the usual operating conditions encountered in the physical refining of crude palm oil.

(refer figure 2).

2. Modelling and Simulation.

There is still restricted access to the latest process design, modelling and simulation technologies. Transfer of technology is only possible with high costs and compliance to the strict patent procedures. In this respect, the research project has manage to obtain and procure strategic softwares to assist in the process design, modelling and simulation of structured packing vacuum distillation plant and operations.

3. Scale-up.

Existing production distillation columns cannot measure performance parameters due to its inherent design and lack of instrumentation. The current project is to develop a laboratory scale experiment rig and a pilot plant rig to fill the gap between basic laboratory scale results and the commercial scale production plant design data.

4. PROCESS DEVELOPMENT AND SCALE-UP.

1 Pilot Plant Experience In Palm Oil Processing.

1. Feasibility Project On Structured Packing Distillation Of Crude Palm Oil.

A feasibility study project was initiated in 1988 onwards culminating in November 1991. The prototype of the pilot plant for palm oil processing is a specially modified glass distillation column in the Chemical Engineering Unit Operations Laboratory. This prototype was used to test the industrial grade borosilicate glass potential. The objectives of this project were:-

- Testing new distillation technologies using standard chemical distillation mixtures to determine the performance and limitations of the structured packings.
- Testing the mechanical and chemical strength of borosilicate glass to endure high temperature of 300 °C, large temperature drop range from 300 °C to 60 °C, extreme low pressure of 10-6 mBar and mechanical shocks due to injection of high pressure steam of 4 Bar into the column under vacuum. These being the conditions of palm oil processes.

(refer figure 3).

2. The scope of the work.

1. The installation, commissioning and testing of a new type of distillation materials i.e. the SULZER MELLAPAK PACKINGS by SULZER CHEMTECH SINGAPORE engineer.
2. The repair and upgrading of the existing equipment and utilities to withstand higher temperatures i.e. from 100 °C to 150 °C and higher vacuum pressures i.e. from 250 mm.Hg(mercury) to 10 mm.Hg.
3. The purchase of new equipments to control the distillation at higher operating conditions.

3. Findings.

SULZER gauze structured packings were installed in the MAKMAL OPERASI UNIT in 1990 and was calibrated with methanol - water mixture. The most critical fault was the vacuum generation and measurement system which limited test to 200 torr pressure.

The conclusions of this experiment was that the first objective were attained successfully in the prototype plant. However, the second and third objectives were unsuccessful due to the extreme temperature and pressure. The manufacturer, CORNING PLC U.K., was contacted on this matter and a new and higher specification glass was recommended for these conditions. This resulted in a new and improved industrial glass design specifications for the purpose of processing palm oil.

As a result of this pioneer venture, NORTON PACKINGS U.S.A. proposed testing their packings at U.T.M. palm oil. A letter of intent has been received and being considered for work in late 1993.

(refer figure 4).

3 Second Scale-up: Laboratory Scale Structured Packings Vacuum Distillation Rig In 1992 - 1993.

The design and fabrication of a lab - scale experimental rig in March, 1993 was to measure the structured packing basic performances at high temperature, low vacuum and stripping agent conditions. This rig extended Hufton & Bravo (1985) work on prediction of structured packing H.E.T.P. It also incorporated Sarkadi (1958) experimental designs for vegetable oil vacuum distillation and SULZER CHEMTECH structured packing pilot plant designs for the measurement of H.E.T.P.

The H.E.T.P. (height equivalent theoretical plate.) parameter was chosen by Hufton & Bravo (1985) work on structured packing performance prediction.This performance parameter was also adopted by the project but the operating conditions and the materials tested are beyond the existing experimental work.

The packed column trouble-shooting phase was completed in April, 1993 with the successful vacuum distillation of 5 percent free fatty acids - palm olein mixture.

(refer figure 5).

5. PROCESS REQUIREMENTS AND DESIGN.

1 Structured Packing Vacuum Distillation Pilot Plant.

This pilot plant experiments will determine whether laboratory scale processes and performance parameters can be scale-up for large scale production.

A laboratory scale experiment rig as described in section 4 is not viable in investigating these aspects as it limited by the:-

- Mass balances errors due to small quantities involved.
- Large heat losses in small diameters columns.
- Uncertainty in forecasting number of theoretical stages.
- Uncertainty of pressured drop due to large wall effects in small columns.
- Different residence times also due to small diameter and length.

2 The Scale Of Equipment and Operations To Be Tested.

- A pilot plant system of at least 50 mm diameter, preferably from 100 mm to 250 mm diameter is suitable to investigate the separation effects, mass transfer effects and hydrodynamics effects involved in the vacuum distillation of high molecular weight mixtures accurately and reliably. (SULZER, LAB & PILOT SCALE DISTILLATION.)

- Data on the use of random and structured packings in high molecular weights mixtures are available on pilot plant scale experiments such as fatty acids distillation using INTALLOX packings. (KISTER, PP.

670..) These can be refer to directly when assessing the H.E.T.P. of crude palm oil.

- All experiment involving structured packings are usually conducted at total reflux. (KISTER, PP. 670..)

- The type of structured packing to be used is the type BX which consist of stainless steel gauze. This packing has a high mass transfer surface area between 500 - 1000 M^2/M^3 as well as a wettability of 100 percent. The typical structured packing used industrially is made of sheet stainless steel but it cannot be made into the smaller dimension of the laboratory - scale distillation of 1 in. inside diameter. Therefore the laboratory - scale distillation will be limited to determining ideal separation and mass transfer performance. However this packing perforations are very small and can be blocked by water droplets. Therefore, water should not be used in the experiment.

(refer figure 6).

3 The Design And Performance Requirements.

1. This pilot plant will be made of industrial grade borosilicate glass. This is a high strength, high temperature resistant and high chemical resistant glass material. Previous pilot plants were constructed of stainless steel. This material has the obvious advantages of flexibility of operating at various pressures, temperatures and chemical corrosion conditions. However, researchers were not able to observe physical

phenomena due to the nature of the material and had to deduce events from measurements.

(refer figure 7).

2. The advantages of glass will be the capability of simultaneously observing and measuring the phenomena hence enabling researcher to deduce its source, its effect on the process and to test means of eradicating it. In short, it would significantly reduce troubleshooting time, effort and costs.

3. Furthermore, glass is transparent to various radiation e.g. infra-red and ultrasonic, and measurements can then made directly. In contrast, stainless steel does allow these radiation to pass except for high energy radiation e.g. gamma rays.

4. It is suggested that for regions exceeding 200 °C in the pilot plant that a new material known as CORBOND which is a composite of fluorocarbon and borosilicate glass is used. This material will be use for the hottest part of the plant i.e. the re boiler which operates at 250 °C. (reference 4.)

5. The rest of the column will be made of thicker glass, 2 mm instead of the usual 1 mm thickness, to withstand the large temperature drop range of 300 °C to 60 °C, extreme low pressure of 10-6 mBar and mechanical shocks due to injection of high pressure steam of 4 Bar into the column under vacuum. (reference 5.)

6. From previous experiences encountered by the manufacturer, large glass plants were not able to guarantee absolute vacuum since a considerable amount of leaks occurred at the joints. These were attributed to unsuitable sealing material in the joints, different expansion due to different materials at the joints e.g. glass - stainless steel joint and vibrations causing displacement of joints. Thus the attainment of vacuum is compromised.

(refer figure 8).

7. Newer joints and sealing were proposed to partially solve the problem. A new joint mechanism made of stainless steel introduced by CORNING in 1991 to link glass parts is proposed to ensure tight fit and still allow for movement between the joints. (reference 5A.)

8. Another improvement is a new locking mechanism based on spring washer to increase tightness of the joint.

9. The last improvement is using VITON instead of TEFLON gaskets since this material is heat resistance and suitable for high vacuum.

10. For heating, SILICONE OILS is used as a heating medium. The heater will be used to initially heat up the medium up to 250 °C and then to maintain this temperature. However, this substance is also toxic since it produced gases and therefore must be isolated.

11. For cooling, water is used as a medium. A chiller will lower the mixture temperature and maintain its cooling temperature.

(refer figure 9).

12. Due to the high risks hazards, most measurements and samples collection must be remote and automated to minimize direct human contact with the pilot plant. This is to ensure safety of the operators as well as to reduce the possibility of human errors e.g. opening of wrong valves, during operations. This means that measurements of pressure, temperature and flowrates are remote, on-line and the smallest volume samples possible extracted using special pumps. These are to ensure minimal pressure losses during sampling.

15. Initial trials on palm oil in 1988 onwards have indicated the long start-up times due to low heating rate and the amount of boil-up rate to sufficiently wet the column internals. It is further proposed that a bypass be build from the boiler to the top of the packing. Hot palm oil will be transferred to the spray feed to pre-wet and accelerate wetting of the packing rather than to wait for sufficient vapour to ascend and do this.

16. Palm oil require new safety procedures in emergency cases. Two outstanding features is that water is never used to put out oil fire since this would cause sprays of burning oil droplets and the other is that the oil must be drained away from the site so as to reduce heat damages.

4 Final Selection Of The Pilot Plant Contractor.

The selected supplier of this pilot plant is Syarikat Production Engineering since it was able to fulfill all the conditions of the purchase and has a long and reliable track record of sales in Malaysia. Furthermore, it has already established direct links with SULZER CHEMTECH

SINGAPORE for the supply of the structured packings. The company was selected due on these factors:-

- The availability of local technical specialists in industrial glass pilot plant construction and installed equipments successfully in Malaysia with accreditions from PETRONAS, SHELL and universities such as U.K.M. and U.S.M. apart from their good track record with U.T.M.
- The company have a sales agreement with the distillation materials supplier, SULZER CHEMTECH SINGAPORE, whom are supplying the structured packings as required by this research project. SULZER CHEMTECH SINGAPORE has no other supplier in Malaysia.
- The company also have process data acquisition and process control equipments and design capability. It also supplies ROSEMOUNT industrial process controls instruments to PETRONAS.
- To engage in transfer of technology such as the supply of appropriate drawings in AUTOCAD compatible files and to develop new product specifications for the glass, gaskets and joints.
- The combined technical experience of CORNING GLASS UK. and SULZER are available. Specifically CORNING GLASS UK. have constructed glass pilot plants with SULZER structured packings and sold several commercial units in Europe.

6. PROCESS MODELLING, INSTRUMENTATION AND ANALYSIS.

1 Application of C.A.D. In Palm Oil Pilot Plant Development.

It is the conclusion of the group at the very inception of the project that AUTOCAD and its enhancements in conjunction with existing process engineering softwares i.e. CHEMSHARE and HYSIM, would enable the realisation of new processes from flow sheet to full scale plant design and to study the implications of the research findings on existing plant designs.

To this end, six process engineering softwares with AUTOCAD linkages have been acquired. Two were procured at research discount prices and four were donated for this project. These are the :-

- CHEMSHARE DESIGN II PROCESS FLOW DIAGRAM package costing $50,000
  (refer figure 10).

- HYSIM GRAPHICAL FLOW SHEET PACKAGE costing $20,000.
  (refer figure 11).

- The AUTOCAD VERSION 11 and the RAPID-PLANT softwares was donated. It is required to convert the process flow sheet from DESIGN P.F.D. and HYSIM to detailed dimensional plant layout, pipings and elevation diagrams for the pilot plant construction.
  (refer figure 12 & figure 13).

- The AUTOCAD HYPERCHEM is another donation and is to model large palm oil molecules and complexes of the palm oil molecules in order to visualise their bond characteristics. These information will be fed into the CHEMSHARE CHEMTRAN properties module in DESIGN P.F.D. to estimate properties of these large molecules.

- The SULPAK Structured Packing Design program is the present tool for SULZER design engineers to design structured packings columns. This was donated to us for this project.
  (refer figure 14).

AUTOCAD drawings of the pilot plant has been prepared for the pilot plant and been transported to the smaller version of AUTOCAD AUTOSKETCH for dissemination. The next step is to re-measure the pilot plant and update the dimensions of the building and plant after installation. These drawings will also be exported to RAPIDPLANT for P.I.D. assignments of the heating, cooling and vacuum piping design schedule in May and June, 1993.
(refer figure 15).

2 Thermal Imaging Of Palm Oil Processes.

In 1990, L.A.S.E.R. group sought the assistance of the thermal imaging research group in P.S.T.P. under En. Mohd Jalis Jelas to investigate the feasibility of using the thermal imaging video camera to survey energy flows in palm oil plants. The result of this endeavor were:-

- A laboratory trial on the visualisation and analysis of thermal energy flows in chemical processes of the Department of Chemical Engineering Process Pilot Plant.
- The visualisation and detection of thermal energy flows and thermal related problems in a Malaysian palm oil refinery in Port Klang.

Both these preliminary studies confirmed the potential applications of this technology for monitoring, analysis and detection of energy flows and energy problems in chemical processes. This endeavour have proved a great success by both U.T.M. and FELDA REFINING. As a result of this survey in 1990-1991, L.A.S.E.R. have formulated a joint research project for 1992 to enhance and develop thermal imaging technology in palm oil process technology.

The present scope of work is to investigate thermal imaging techniques for visualisation and analysis of gas-vapour-liquid flows in pilot plant distillation. The thermal imaging system will use to visualise the temperature profiles occurring in a borosilicate glass pilot plant scale vacuum distillation unit in U.T.M. Semarak. During distillation, the vapour and liquid phases of palm oils and fatty acids flow within the structured packings and induce mass transfer. However due to changes in their physical properties during the distillation, these flows are disturbed. In ideal mixing flows, the distribution of temperatures are uniform. In the event of disturbances, the flows are disturbed and the temperature profile are altered as a result. The system would give an overall as well a specific view of the temperature profile in the column and locate cold and hot spots i.e. areas of disturbances.

7. CURRENT AND FUTURE PROGRESS.

1 Existing Pilot Plant Status.

- Silicon Oil Heater System.
- Vacuum Generation & Measurement System. *(refer figure 16).*
- Glass Condenser And Cooling Water System. *(refer figure 17).*
- Power Back-up System.
- Structure, Platform And Lifting System. *(refer figure 18).*
- Pilot Plant Building. *(refer figure 19).*

2 New Experimental Designs and Problems.

1. Design Of Nitrogen Injection And Control System. *(refer figure 20).*

2. Design Of Rapid Fatty Acids Determination In Palm Oil System.

3. Design Of Structured Packing On-line Temperature Measurement System.

4. The Condenser Sub cooling effect.

It is found that the condenser cooling water temperature was too low at 15 °C and this causes sub-cooling of the fatty acids vapour leading to solidification in the condenser. A new cooling water temperature and heat transfer coefficient is being determined from laboratory rig experiment to overcome this problem.

5. Distillation Heat Losses.

High heat transfer losses occurred between the re boiler and the condenser. This slows the vapour flow to the top of the column. Two insulators design are being proposed; The passive and active insulator design. Initial priority is on the passive insulator to be build by Yahya.

6. Distillation Liquid bottom sampling and measurement unit.

The high vacuum in the column requires that a pressure lock system to obtain liquid sample. The sample must be extracted from the column, cooled to liquid form and de-pressurised for removal. A prototype is has been fabricated and undergoing test in the laboratory rig.

8. CONCLUSION.

It is in the interest of the nation to develop the means of measuring and determining processes performance followed by the designing of the enhancement of the vacuum distillation plant. This is so that Malaysian refiners are able to made the best decisions on future process retrofits. The best retrofit will ensure that Malaysian palm oils are able to compete in the global edible oils market in the future.

NOTE.

REPORT STRUCTURE.

Develop palm oil sampling and analysis procedures.

PORIM palm oil FFA analysis laboratory test using titration.

Palm oil FFA analysis laboratory test using automatic titration.

Palm oil FFA analysis laboratory test using UV spectrophotometer.

Develop physiochemical properties determination and measurement rigs.

Boiling points of unrefined palm oils.

Surface Tension measurement procedures w.r.t. temperature and composition.

Develop process model, simulation and design computer systems.

Computer data acquisition system for temperature and pressure.

Inter-plate temperature measurement system.

Acquisition of SULZER structured packing design computer software.

Acquisition of AUTOCAD computer software for pilot plant design.

Acquisition of AUTOCAD HYPERCHEM computer software for molecular modeling.

Develop laboratory and pilot plants.

Laboratory scale structured packing vacuum distillation column for palm oil to determine HETP due to physio-chemical properties, temperature and pressure effects.

Pilot plant scale structured packing vacuum distillation column for palm oil to determine HETP due to scale-up effects.

Pilot plant distillation silicon oil heating system.

Pilot plant distillation vacuum system.

Pilot plant distillation condenser system.

Pilot plant structure, platform and winch equipments.

Distillation distillate and re boiler liquid sampling system.